1,079,388

PATENT SPECIFICATION

NO DRAWINGS

1,079,388

Date of Application and filing Complete Specification: Oct. 5, 1965.

No. 42313/65.

Application made in United States of America (No. 410,288) on Nov. 10, 1964.

Complete Specification Published: Aug. 16, 1967.

Index at acceptance:—C5 D(6A2, 6A5A, 6A5B, 6A5C, 6A5D1, 6A5D2, 6A5E, 6A5F, 6A8B, 6B1, 6B2, 6B3, 6B4, 6B7, 6B10B, 6B12K1, 6B12K2, 6B12K3, 6B12M, 6B12M1, 6B12M2, 6B12N3, 6B12N4, 6B12N5, 6B13, 6B15, 6C6)

Int. Cl.:—C 11 d 1/72

COMPLETE SPECIFICATION

Detergent-Softener Composition

ERRATA

SPECIFICATION No. 1,079,388

Page 2, line 9, *for* "taker" *read* "taken"
Page 3, line 20, *for* "suitable" *read* "suitably"
Page 3, line 31, *for* "trihydroxyethyldodecyl" *read* "trihydroxyethyl dodecyl"

THE PATENT OFFICE
12th September 1967

ing agents, optical brighteners and germicidal agents.

It has now been discovered that a detergent-softener composition capable of washing and simultaneously softening textiles may be produced from a composition which comprises a nonionic detergent, a cationic fabric softener, and a nonionic soil-suspending agent, said detergent and said fabric softener being present in a ratio by weight of between 0.5 : 1 and 2.5 : 1.

A nonionic detergent composition which can be used in the formulation of this invention consists essentially of a condensation product of polyethylene oxide in which the polyethylene oxide chain acts as the hydrophilic group and the hydrophobic group is derived from such materials as fatty acids, alcohols (including fatty alcohols), mercaptans, amines, amides, substituted phenols and glucosides. By using combined oxyalkylation with ethylene oxide and propylene oxide, compounds with "tailor-made" hydrophilic-hydrophobic balances can be produced. The preparation of basic nonionic detergents is shown in U.S. Specifications Nos. 1,970,578 and 2,213,477. These substances are also disclosed in the article by Jelinek and Mayhew under the title "Nonionic Surfactants" published in the Textile Research Journal, Volume 24, Number 8, August, 1954.

Fabric softeners are essentially lubricants of highly spreading and penetrating power and can be divided into two groups, a non-substantive class and substantive class. The non-substantive members are either anionic or nonionic in nature and the substantive class members are cationic. The fabric softeners used in the formulation of this invention are taken from the substantive or cationic class of softening agents

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Int. Cl.:—C 11 d 1/72

COMPLETE SPECIFICATION

Detergent-Softener Composition

We, GENERAL FOODS CORPORATION, a Corporation organized under the laws of the State of Delaware, United States of America, of 250 North Street, White Plains, State of New York, United States of America, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to detergent compositions, and more particularly, to a germicidal detergent-softener composition containing inorganic alkali metal salt builders, nonionic soil-suspending agents, and optical brighteners.

The incompatability of detergents and fabric softeners in laundering compositions has been known for a long time and has necessitated the separate addition of a softening agent during the rinse portion of the washing cycle. In the case of anionic detergents the softener reacts with the detergent to form a greasy, insoluble scum while with nonionic detergents (low sudsers) the softener is not found to last through the wash and rinse cycles in enough quantity to add-on to the fabric being washed. Even when the softener is applied during the rinse cycle, problems have been presented due to the presence of residual amounts of detergent in the textile material.

Therefore, it would be highly desirable to develop a formulation capable of simultaneously acting as a detergent and a softener which would obviate the need for interrupting the normal wash cycle and separately adding as a second ingredient the softening agent during the rinse portion of the washing cycle. A detergent composition having a built-in fabric softener and soil-suspender would allow textiles to be washed and softened at the same time and could also include compatible bleaching agents, optical brighteners and germicidal agents.

It has now been discovered that a detergent-softener composition capable of washing and simultaneously softening textiles may be produced from a composition which comprises a nonionic detergent, a cationic fabric softener, and a nonionic soil-suspending agent, said detergent and said fabric softener being present in a ratio by weight of between 0.5 : 1 and 2.5 : 1.

A nonionic detergent composition which can be used in the formulation of this invention consists essentially of a condensation product of polyethylene oxide in which the polyethylene oxide chain acts as the hydrophilic group and the hydrophobic group is derived from such materials as fatty acids, alcohols (including fatty alcohols), mercaptans, amines, amides, substituted phenols and glucosides. By using combined oxyalkylation with ethylene oxide and propylene oxide, compounds with "tailor-made" hydrophilic-hydrophobic balances can be produced. The preparation of basic nonionic detergents is shown in U.S. Specifications Nos. 1,970,578 and 2,213,477. These substances are also disclosed in the article by Jelinek and Mayhew under the title "Nonionic Surfactants" published in the Textile Research Journal, Volume 24, Number 8, August, 1954.

Fabric softeners are essentially lubricants of highly spreading and penetrating power and can be divided into two groups, a non-substantive class and substantive class. The non-substantive members are either anionic or nonionic in nature and the substantive class members are cationic. The fabric softeners used in the formulation of this invention are taken from the substantive or cationic class of softening agents

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widely used in the textile industry to improve the "hand" and drape properties of a fabric. The softeners, which are cationic surface active agents dissolve or disperse in water, concentrate and orient at interfaces from their aqueous dispersions, and ionize. Preferably, the surface active agent is such that the cation includes a hydrophobic hydrocarbon chain containing from 8 to 25 carbon atoms. Preferably, the softening agent will be any one of the conventional quaternary ammonium salt type of softeners used in the laundering and textile industry, e.g., distearyl dimethyl quaternary ammonium chloride or ditallow dimethyl quaternary ammonium methosulfate. Other softeners which may be used are taker from the following classes of compounds: amino esters, carbonates, tertiary amine , amidoamines, phosphonium compounds, benzimidazoles, biguanidines, and imidazolines or glyoxaldines. The softener compound may be combined with various wetting agents to enhance its ability to add-on to the fabrics being laundered.

The soil-suspending agent used in the detergent-softener composition should be ninionic in nature and can be present in amount of from 0.05 to 2.0% by weight. Polyvinylpyrrolidone (PVP) is preferred as the soil-suspender, typically this agent should have an average molecular weight of from 15,000 to 40,000 and should be water soluble to an extent of from 0.001% and 0.1%. The properties of PVP as a soil-suspender in a detergent are shown in U.S. Specification No. 3,000,830. Other nonionic soil-suspenders are polyvinyl alcohol, polyvinyl acetate, and certain colloidal gums such as methylcellose.

Since detergency (soil removal) and softening (add-on) are diammetrically opposing actions the ratio between the nonionic detergent and the softener is critical. It has been found that the optimum and therefore preferred weight ratio of detergent to softener is from 1.2:1 and 1.6:1, preferably 1.4:1. At greater than 1.6:1 the detergent tends to wash out the softener before it has a chance to deposit on the fabric. This softener wash out is gradual and the softening activity is continually noticed, even though gradually decreasing, by careful evalution of the "hand" or softening effect. Below 1.2:1 the softener effect begins to exceed the detergent effect. This action also occurs slowly and is noticeable by careful measurement of the soil removal of standard soil cloth in a terg-o-tometer and subsequent measurement on a Hunter Color Difference Meter. Below 1.2:1 the softener tends to overwhelm the detergent and soil removal ability is rapidly lost. The operable limits of detergent to softener have usually been found to be between 1:1 and 1.9:1. Below the limit of 1:1 soil removal is often so slight as to be of no practical value for laundering purposes. Above the limit of 1.9:1 the detergent action may be so strong that it eliminates any softening effect on the fabrics being washed. However, with careful choice of detergent and softener the broadest limit of 0.5:1 to 2.5:1 is available.

The detergent-softener composition of this invention may also include inorganic alkali metal builders, a corrosion inhibitor, a germicidal agent of the quaternary ammonium salt type, optical brighteners, and bleaching agents.

A specific detergent formulation of this invention can be represented as follows:

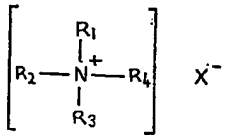
	Percent by weight
Inorganic builders	55.0
Moisture	15.0
Nonionic detergent	11.0
Fabric softener	8.0
Corrosion inhibitor	6.3
Germicide	3.0
Nonionic soil-suspender	1.5
Optical brightener	0.2
	100.0%

Among the detergent builders or fillers which may be used are the alkali metal salts of inorganic acids. These builders sometimes may contain strong bases or neutral salts. A heavy duty type detergent may contain more free alkali while a similar formulation for light duty may contain a neutral salt such as sodium sulfate. Specific builders or fillers which can be used for this invention are soda ash, borax,

sodium metasilicate, sodium sulfate, sodium hexametaphosphate, trisodium phosphate, tetrasodium pyrophosphate, sodium acid pyrophosphate, sodium tripolyphosphate, sodium monobasic phosphate, sodium dibasic phosphate, sodium carbonate, and sodium acid carbonate. The range of these ingredients may vary from 20 to 80% by weight of the total detergent composition. The amount of nonionic detergent and softener for a formula containing builders and fillers will be from 5 to 25% for the nonionic detergent and from 2 to 15% for the softener.

The corrosion inhibitor used in the formulation of this invention may be any conventional alkali metal silicate inhibitor such as sodium silicate or sodium metasilicate. Without silicates any phosphate detergent builders used in the formula will attack the steel and aluminum parts of the washing machine and rapidly corrode these parts. The amount needed is dependent upon the amount of polyphosphate used. About 8% of sodium silicate is enough to carry about 40% polyphosphate. The silicate inhibitors also have some soil removal and soil suspending properties. The amount of corrosion inhibitor in the final detergent formula is usually between 6 to 12% and the amount of sodium tripolyphosphate is 20 to 55%. The amount of a filler, such as sulfate, usually used together with the polyphosphate builders is 5—25%.

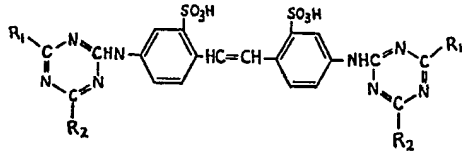
A cationic germicidal agent may also be included in the detergent-softener composition of this invention. This ingredient can most suitable be an aliphatic quaternary ammonium type salt having a general formula as follows:



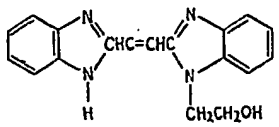
wherein X represents a sulfate or halogen radical, R_1, R_2 and R_3 are aliphatic radicals having from 1—7 carbon atoms, and R_4 is an aliphatic radical having from 8—18 carbon atoms. Examples of quaternary ammonium salts which may be used are: trimethyl dodecyl ammonium bromide, allyl dibutyl dodecyl ammonium bromide, methyl diallyl dodecyl ammonium iodide, diethyl acetonyl dodecyl ammonium chloride, diethyl (beta-hydroxy gamma-butyloxypropyl) dodecyloxymethyl ammonium chloride, diethyl dodecyl geranyl thioethyl ammonium bromide, dibutyl allyl dodecyloxymethyl ammonium chloride, dimethyl geranyl dodecyl ammonium chloride and trihydroxyethyldodecyl ammonium bromide. Anionic germicidal agents should be avoided since such agents will react with the cationic softener. The cationic germicide is present in an amount of from 0.01 to 5.0% depending on the biocidal activity of the particular agent and the use intended for the final detergent formulation.

The formulation of this invention may also contain optical brighteners and bleaching agents. Only a limited number of optical brighteners have been found effective in the detergent-softener composition of this invention. Most anionic brighteners are not suitable since they react with the cationic softener or cationic germicide to give optically inactive compounds. Most nonionic or cationic brighteners are unstable in alkali or fail to add-on to the fabric and are washed away. Also, the brightener should preferably be hypochlorite stable and not react with any bleaching agents which are blended as part of the formula or added separately by the consumer during washing.

Brighteners which are compatible with the detergent-softener composition include a stilbene triazine derivative having the general formula:



wherein R_1 is monoethanolamine and R_2 is diglycolamine; and a benzimidazole derivative such as 1 - benzimidazole - 2 - (N - hydroxyethyl) - benzimidizole ethylene having the following formula:



These brighteners can be used in combination or alone. The stilbene triazine derivative is used in an amount of from 0.001 to 0.2% by weight while the benzimidazole derivative can be present in an amount of from 0.001 to 3.0% by weight.

Bleaching agents suitable for incorporation into the detergent-softener formulation include any bleach taken from the class of alkali metal hypochlorites and alkali metal perborates.

This invention will now be described by reference to several specific examples which show different embodiments of a detergent-softener composition in accordance with this invention.

In producing the detergent-softener formulation of this invention, the various ingredients are mixed with water to form a slurry and then spray-dried. The slurry usually has a concentration of 55% to 60% by weight solids prior to spray-drying and is dried to about 10% to 15% by weight moisture content. In making up the slurry the corrosion inhibitor (sodium silicate) is first added to water (at 70° F.), then the phosphate builders are added, next the filler (sodium sulfate), softener, brighteners, germicide and nonionic detergent are added with the soil suspending agent (PVP) be added last. The product is then cooled and packaged.

This invention will now be described by reference to several specific examples which show different embodiments of a detergent-softener composition in accordance with this invention.

EXAMPLE 1

Detergent Formula	Parts by Weight
Sodium tripolyphosphate	40.0
Sodium sulfate	13—36
Moisture	15.0
Sodium silicate	8.0
Decyl dimethyl benzyl ammonium chloride	3.0
Polyvinyl pyrrolidone	1.5

Between 15 and 32 parts (by weight of the total detergent formula) of different mixtures of nonionic detergent and cationic softener were blended into the above formula. The ratio of nonionic detergent to softener was varied within the operable limits of this invention and the softening and soil removal properties of each formulation was measured. The parts by weight of sodium sulfate were varied to maintain a constant total formula concentration in the wash during these tests. The parts by weight of the remaining ingredients were kept constant regardless which detergent-softener formula was being tested. The results are shown in following table:

Detergent-Softener System	Parts by Weight	Weight Ratio Detergent/ Softener	Softness Rating	Units Soil Removal
Polyoxyethylene Ether of dodecylphenol ("Sterox"DJ) and ditallow dimethyl quaternary ammonium methosulfate	10.9/21.8	0.5/1	4.0	8.0
	10.9/7.8	1.4/1	3.0	8.5
	10.9/4.3	2.5/1	1.0	9.3
Polyoxyethylene ether of tall oil ("Sterox CD") and ditallow dimethyl quaternary ammonium methosulfate	10.9/21.8	0.5/1	4.0	4.1
	10.9/7.8	1.4/1	3.5	4.4
	10.9/4.3	2.5/1	2.0	4.4
Polyoxypropylene polyoxyethylene glycol ("Pluronic" L44) and ditallow dimethyl quaternary ammonium methosulfate	10.9/21.8	0.5/1	3.5	3.2
	10.9/7.8	1.4/1	3.5	4.4
	10.9/4.3	2.5/1	2.0	4.8
Polyoxyethylene ether of nonyl phenol (Sterox NJ) and ditallow dimethyl quaternary ammonium methosulfate	10.9/21.8	0.5/1	4.0	7.9
	10.9/7.8	1.4/1	3.5	8.4
	10.9/4.3	2.5/1	1.5	9.1
Polyoxyethylene ether of isooctylphenol ("Triton" X-100) and ditallow dimethyl quaternary ammonium methosulfate	10.9/21.8	0.5/1	4.5	4.0
	10.9/7.8	1.4/1	3.5	4.4
	10.9/4.3	2.5/1	3.0	4.7
Polyoxyethylene ether of dodecyl phenol ("Sterox" DJ) and distearyl dimethyl quaternary ammonium chloride	10.9/21.8	0.5/1	4.0	8.0
	10.9/7.8	1.4/1	3.0	8.5
	10.9/4.3	2.5/1	1.0	9.3
Polyoxyethylene ether of nonyl phenol ("Sterox"NJ) and distearyl dimethyl quaternary ammonium chloride	10.9/21.8	0.5/1	4.0	7.9
	10.9/7.8	1.4/1	3.5	8.4
	10.9/4.3	2.5/1	1.5	9.1

The words "Sterox", "Pluronic" and "Triton" are registered Trade Marks.

Soil removal ability and softening ability of the above formulations were tested by known procedures. For the soil removal rating a standard soil cloth from U.S. Testing Corporation was used and soil removal was measured on a Hunter colorimeter. For the softness rating washed terry cloth was rated by hand using a scale of 5 for soft and 1 for harsh. Several washings were conducted, the towels spun-dried and then line dried at 70°F. and 50—60% relative humidity. The towels used were 42" × 24" and enough were used to form a washload of six pounds. The mean score of all the ratings for each sample was reported. Harshness control (zero rating) was set up by washing new terry cloth towels (42" × 24") at 128°F. in a mixture of 20 parts dodecyl benzene sulfonate and 60 parts sodium tripolyphosphate. The towels were allowed to line dry over 24 hours at 70°F. and 50—60% relative humidity. Softness control (5 rating) was prepared by rinsing one set of previously harshened towels with a 6% solution of ditallow dimethyl quaternary ammonium

chloride and then allowing the towels to line dry (at 70°F. and 50—60% relative humidity).

Test washings for softness were done in a standard home laundry washing machine (RCA Whirlpool) at a washwater temperature of 128°F. and for soil removal in a U.S. Testing Company Tergo-O-Tometer at 130°F. The complete formulation was used at a solution strength of 0.21% or 116 grams of formula per washload.

As can be seen by reference to the above table, optimium softness and soil removal was attained at a detergent-softener ratio of 1.4:1. However, some cooperative softening and detergent action was still observable at the outside limits of 0.5/1 and 2.5/1. It can be seen that at the detergent/softener ratio of 0.5/1 the softening action begins to predominate and the detergent action is rapidly lost while at 2.5/1 the detergent is found to be too strong to allow any appreciable softening.

EXAMPLE 2

The above procedure was repeated with the exception that a bleach stable optical brightener ("Uvitex" SIA Conc., manufactured by Ciba Chemical Company: the word "Uvitex" is a registered Trade Mark) was incorporated at a level of 0.1% by weight of the total detergent formula.

The use of this brightener was found to overcome a yellow-type of tinge left in towels washed according to the procedure of Example 1. However, the towels still had a greenish or neutral white tinge.

EXAMPLE 3

The procedure of Example 2 was repeated with the exception that 2.5% by weight of Arctic White CK brightener containing 20% active ingredient (manufactured by Hilton-Davis Corporation) was blended into the detergent formula. This formulation was found to change the final color of the washed fabric from a greenish or neutral white hue to a more desirable reddish white hue.

WHAT WE CLAIM IS:—

1. A detergent composition consisting essentially of nonionic detergent, a cationic fabric softener, and a nonionic soil-suspender, said detergent and said softener being present in the ratio by weight of from 0.5:1 to 2.5:1.

2. A composition according to claim 1, wherein the ratio of detergent to softener is from 1.2:1 to 1.6:1.

3. A composition according to either of claims 1 and 2, wherein the nonionic detergent is a condensation product of polyethylene oxide.

4. A composition according to claim 3, wherein the nonionic detergent is a polyoxyethylene ether of an alkyl phenol.

5. A composition according to claim 4, wherein the alkyl phenol is a nonyl or a dodecyl phenol.

6. A composition according to any one of claims 1 to 5, which contains 5 to 25% by weight of nonionic detergent.

7. A composition according to any one of claims 1 to 6, wherein the cationic fabric softener is a quaternary ammonium salt.

8. A composition according to claim 7, wherein the softener is an alkyl dimethyl quaternary ammonium chloride.

9. A composition according to claim 8, wherein the softener is ditallow dimethyl quaternary ammonium methosulfate.

10. A composition according to any one of the preceding claims, which contains 2 to 15% by weight of softener.

11. A composition according to any one of the preceding claims in which the soil-suspender is polyvinylpyrrolidone.

12. A composition according to any one of the preceding claims which contains from 0.05 to 2.0% by weight of soil-suspender.

13. A composition according to any one of the preceding claims, which contains from 20 to 80% by weight of inorganic builders.

14. A composition according to claim 13, which contains from 20 to 55% by weight of inorganic alkali metal phosphate builders, and 1—25% by weight of inorganic filler.

15. A composition according to claim 14, which contains from 20 to 25% by weight of inorganic alkali metal phosphate builders.

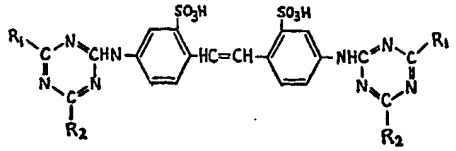
16. A composition according to any one of the preceding claims which contains from 0.01 to 5.0% by weight of an aliphatic quaternary ammonium germicide.

17. A composition according to claim 14, or either one of claims 15 and 16 as dependent on claim 14 which contains 6 to 12% by weight of corrosion inhibitor.

18. A composition according to claim 17 in which the corrosion inhibitor is an alkali metal silicate.

19. A composition according to any one of the preceding claims which contains an optical brightener.

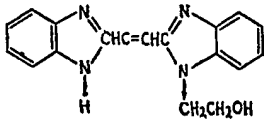
20. A composition according to claim 19, in which the optical brightener is from 0.001 to 0.2% by weight of a stilbene triazine optical brightener having the following formula:



wherein R_1 is monoethanolamine and R_2 is diglycolamine.

21. A composition according to claim 19, in which the optical brightener is from 0.001 to 3% by weight of a benzimidazole derivative.

22. A composition according to claim 21 in which the benzimidazole has the formula:



23. A composition according to any one of the preceding claims which contains from 10 to 15% by weight moisture.

24. A detergent softener composition substantially as hereinbefore described with particular reference to the Examples.

STEVENS, LANGNER, PARRY & ROLLINSON
Chartered Patent Agents,
Agents for the Applicants.

Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press. —1967. Published by The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.